Determination of Formaldehyde and VOCs in Wood-based Products using an Automated Micro-Scale Chamber

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Abstract

An automated micro-scale chamber method based on 2,4-dinitrophenylhydrazine (DNPH) derivatization and Tenax TA adsorption, for sampling formaldehyde, acetaldehyde, acetone, acrolein, as well as volatile organic compounds (VOCs) was developed. Following the sampling step, the Tenax TA tubes were immediately transferred to a thermal desorption (TD)-GC/MS system for analysis. The DNPH tubes were transferred to an autosampler tray for temporary storage and later eluted and analyzed by high performance liquid chromatography (HPLC) using a diode array detector (DAD).

Different wood-based products were analyzed for their emissions of the target compounds. Different emission values of formaldehyde, acetaldehyde and acetone were obtained, no acrolein was detected in any of the samples. The hexanal emission values obtained using the DNPH-HPLC method and Tenax-GC method were compared. The results showed that using the DNPH-HPLC method for determination of hexanal concentrations in chamber air resulted in analyte discrimination with lower emission values being reported. Finally, the long term emission behavior of a medium-density fiber board (MDF) was determined based on both DNPH-HPLC-DAD and Tenax TA-TD GC/MS analysis.

Introduction

Wood-based products, such as particle board, oriented-strand board (OSB), high-density fiber board (HDF), medium-density fiber board (MDF) and plywood are widely used indoors in buildings and they frequently contain formaldehyde-based adhesives. For this reason, wood-based products are generally a major source of formaldehyde in indoor air along with materials such as flooring, insulation and coating [1].

Exposure to formaldehyde in air can cause irritation of the mucous membranes. Formaldehyde is classified as a probable human carcinogen (EPA 1991) and a suspected carcinogen (European Union 2004). The World Health Organization (WHO) has recommended that formaldehyde concentrations be kept below 100 µg/m³ even for a half hour short-time exposure in order to prevent nose and throat irritation (1987). This value (100 µg/m³) is used as reference in countries such as Germany, the U.K. (0.5 h), Norway (0.5 h) and China (1 h) [1]. Trapping of formaldehyde and other volatile aldehydes and ketones on DNPH impregnated sorbents with subsequent HPLC-DAD determination is widely used in international standards [2, 3].

In addition to the very volatile organic compounds formaldehyde, acetaldehyde and acrolein, VOCs ranging from n-hexane to n-hexadecane are emitted from wood-based products and have an impact on the indoor air quality as well as on the health of building occupants. According to the German AgBB scheme, the total VOC (TVOC) values after 3 days in an environmental test chamber should meet the criteria ≤ 10 mg/m³ and after 38 days ≤ 1.0 mg/m³ [4]. For each individual VOC , the lowest concentration of interest (LCI) is specified [4]. For determination of VOCs in emission test chamber, active sampling using Tenax TA Tube followed by thermal desorption - GC-MS/FID is widely used [5].

Environmental test chambers are widely used to determine emission values of building materials and other products that come into contact with indoor air. These chambers are normally operated off-line. The specimen is put into a closed chamber under specified temperature and relative humidity conditions. Into the chamber, pure air or inert gas is supplied at a required air exchange rate. After specified time periods, air samples are collect-

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ed by manually placing a Tenax TA sorbent tube and/or DNPH cartridge into the outlet flow and sampling a specified volume at a specified flow using a pump. Afterwards, the loaded Tenax TA sorbent tube and/or DNPH-cartridge are removed and placed in individually sealed storage containers for later analysis.

In this study, an automated micro-scale chamber method based on the GERSTEL DHS Large autosampler (Figure 1) was developed, which offers a simple-to-use efficient screening method for material emission testing. DHS Large enables analysis of heterogenous or bulk samples with a volume up to 1 L without the need for cutting, grinding or other means of homogenizing normally used in order to take a smaller representative sample. The DHS L sample containers are made of stainless steel with an inert coating. The vessels are air tight and exhibit very low VOC/SVOC background values. Different types of inlet gases can be selected, such as synthetic air, nitrogen or helium to name the most widely used. A controlled purge flow from 5-100 mL/min results in an air change rate from 0.3 - 6/h when a one liter container is used. The DHS Large autosampler provides 11 sample positions and the PrepAhead function enables efficient parallel processing: During the ongoing GC/MS analysis the next sample is extracted in the DHS L autosampler for efficient sample throughput and GC/MS instrument utilization.

Figure 1: DHS-Large autosampler.

Experimental

Materials

For HPLC-DAD calibration, a standard mixture of aldehydes/ketones-DNPH TO11/IP-6A was obtained from Sigma Aldrich. ORBOTM-DNPH tubes (LpDNPH on silica gel, 120 mg) were purchased from SUPELCO. These are 90 mm length glass tubes with a frangible seal at one end. The tubes contain a 120 mg bed of DNPH-coated silica gel and both ends are sealed with PTFE caps to avoid air ingress and contamination. Before use, the PTFE caps are removed and the frangible end is broken.

For micro-scale chamber-DNPH-HPLC-DAD system calibration, an aqueous mix of formaldehyde and acrolein was prepared, the formaldehyde solution (37 wt. % H_2O, contains 10-15 % methanol) and acrolein (≥ 99.0 %) were purchased from Sigma Aldrich. This additional complete system calibration was performed due to the well-known formation of multiple derivatization products of acrolein [3].

For Tenax-TD-GC/MS calibration, a mixture of pentanal, toluene, hexanal, 2-hexanoic acid, octanal, and dodecane was made, the individual standards were purchased from Sigma Aldrich. Tenax® TA tubes were obtained from GERSTEL GmbH & Co. KG. Different wood-based samples were used: oriental-strand board (OSB); medium-density fiber board (MDF); and plywood. All samples were cut to 6 cm x 9 cm size with front and back surfaces exposed to the chamber and all edges covered with aluminum foil. In addition, several wood toys were weighed and tested including puzzle pieces and wood blocks. All samples were placed vertically in the chamber.

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Micro-scale chamber method

DHS Large method parameters used for the micro-scale chamber analysis are shown in Table 1. A surface specific air flow (q) of 0.2778 m³/m²h was chosen following the AgBB specification, which requires an air exchange rate of 0.5/h and a loading factor of 1.8 m²/m³ for wall, flooring and ceiling area [4]. The loading factor and air exchange rate can be varied as long as the surface specific air flow for the product type is met [6]. Since a one liter container is used, an air exchange rate of 0.5/h results only in a flow rate of 8.3 mL/min, which is too small for the method, so the air exchange rate was varied to 3/h for the micro-scale chamber. To achieve the same surface specific air flow, a specimen size of 108 cm² was analyzed. For dual side exposure, each side must be 54 cm², therefore a sample size of 6 cm x 9 cm was chosen. Several different surface specific air flows are recommended in different standards methods. The AgBB recommendation is 0.2778; in ASTM D6007-14, different surface specific air flows are recommend, for example, 1.905 for MDF, 1.172 for particleboard flooring panels, industrial particleboard panels, industrial hardwood plywood panels, and 0.526 for hardwood plywood wall paneling [6]. In this study, we followed the AgBB recommendation, the goal was to use the same chamber parameters for quantitative comparison of different wood-based samples, not to achieve the same emission values obtained from standard-sized, large emission chambers. In contrast to standard emission chambers no humidification of the purge gas was used.

For formaldehyde, acetaldehyde, acetone and acrolein, a three liter air volume was sampled on ORBOTM-DNPH tubes followed by elution with 2 mL acetonitrile and HPLC-DAD analysis. For other VOCs 500 mL air volumes were sampled on Tenax TA-filled tubes followed by thermal desorption-GC-MS analysis. Between each sampling, an empty tube was inserted to simulate emission chamber conditions. The tube exchange resulted in a short interruption of the gas flow. A schematic diagram of the automated micro-scale chamber sampling process in the DHS Large system is shown in Figure 2. Please note: In this study, elution of the DNPH derivatives into an open vial was performed manually under nitrogen atmosphere, no syringe filter and needle was used. A comparison of results obtained with and without syringe filter indicated that the filter is not needed, a pre-column provided sufficient protection of the analytical column.

Table 1: DHS Large parameters for the micro-scale chamber method.

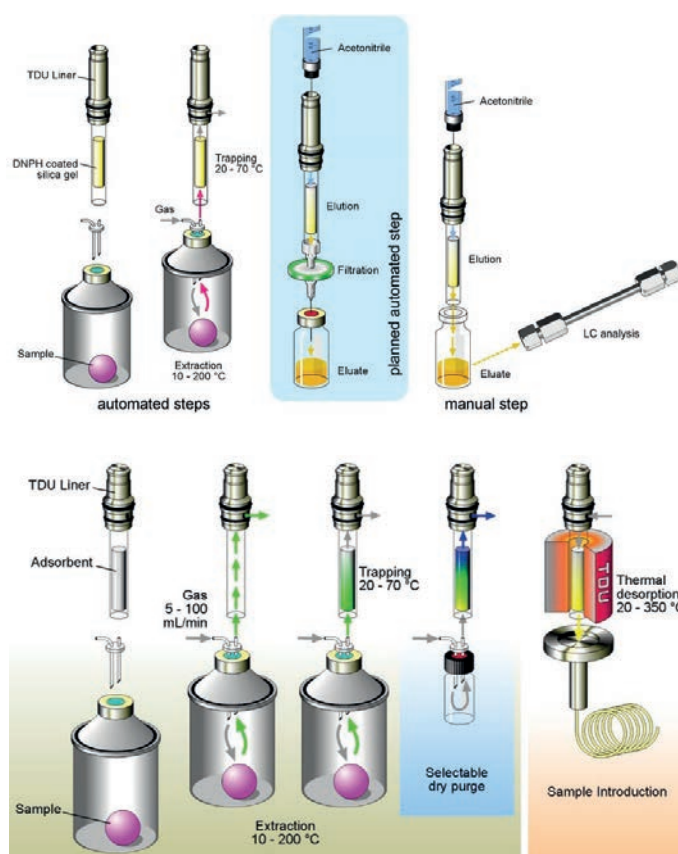| Micro-scale chamber parameter	Setting
Loading factor L [m²/m³]	10.8
Air exchange rate n [h⁻¹]	3
Surface specific air flow q [m³/m²h]	0.2778
Chamber volume V [m³]	0.001
Sample surface A [m²]	0.0108
Temperature [°C]	23 ± 1
Relative humidity [%]	0

Figure 2: Schematic diagram of DNPH sampling (top) and Tenax sampling (bottom) using DHS Large as a micro-scale chamber.

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Analysis conditions DNPH-HPLC-DAD

DNPH elution	2 mL Acetonitrile
HPLC column	Nucleoshell RP 18plus
	50 mm x 2 mm x 2.7 µm
	(Macherey Nagel)
Pump	gradient,
	flowrate = 0.8 mL/min
Mobile phase	A - water, B - acetonitrile
Gradient	Initial 20 % B
	15 min 60 % B
Injection volume	5.0 µL (full loop injection)
Detector	1200 SL HPLC-DAD
	(Agilent Technologies)
	360 nm, bandwidth 30 nm

Analysis conditions Tenax-GC-MSD

TDU	20°C; 720°C/min; 275°C (5 min)
Column	30 m Rxi-5ms (Restek)
	d_i = 0.25 mm d_f = 0.5 µm
Pneumatics	He, constant flow = 1.5 mL/min
	split 1:5

Oven	40 °C (4 min); 5 °C/min to
	90 °C; 8 °C/min to
	200 °C; 20 °C/min to
	280 °C (4 min)
Detector	5973 MSD (Agilent Technologies)
	Scan, 33 - 300 amu

Results and Discussion

Linearity, LOD, LOQ. Using HPLC-DAD, good linearity was obtained for aldehyde/ketone-DNPH derivatives in the range from 0.01 to 15 µg/mL with R² larger than 0.9991. Formaldehyde, acetaldehyde and acetone were found to have unavoidable blank values caused by the DNPH sorbent tube (background per tube: formaldehyde < 25 ng, acetaldehyde < 35 ng, acetone < 120 ng) as well as the collection chamber and the elution process due to contact with laboratory air. Therefore, the blank value method according to DIN 32645 was used to determine the limits of detection (LOD) and limits of quantification (LOQ).Three liter samples of air were collected from an empty DHS Large container through DNPH tubes. Five blank samples were run using five different DHS Large containers and five ORBOTM-DNPH tubes (Table 2).

Table 1: Linearity of liquid injection of an aldehyde/ketone-DNPH standard and LOD and LOQ of the micro-scale chamber-DNPH-HPLC-DAD method (3 L sampling, 2 mL ACN) according to DIN 32645.

Aldehyde/Ketone-DNPH	Linear range [µg/mL]	Linearity (R²)	LOD [µg/mL], (n=5)	LOQ [µg/mL], (n=5)
Formaldehyde-DNPH	0.01 - 15	1.0000	0.010	0.032
Acetaldehyde-DNPH	0.01 - 15	0.9995	0.041	0.123
Acetone-DNPH	0.01 - 15	0.9998	0.504	1.511
Acrolein-DNPH	0.01 - 15	0.9995	n.d.	n.d.
Others-DNPH	0.01 - 15	> 0.9991	n.d	n.d.

Since the calibration standards contain the DNPH derivatives of aldehydes and ketones, the LODs and LOQs need to be recalculated to the underivatized compounds taking into account the molecular weight of the respective molecules (Figure 3). Furthermore, liquid concentrations of aldehydes/ketones need to be converted into air concentrations for the micro-scale chamber-DNPH-HPLC-

DAD method based on a 3 liter sampling volume and an ACN elution volume of 2 mL (equation 1 and table 3). The LOQs were as follows: formaldehyde: 3.05 µg/m³; acetaldehyde: 14.8 µg/m³; and acetone: 246 µg/m³. The high LOQ for acetone was caused by the high background concentration in the laboratory air.

$$\text{Air concentration } [\mu g/m^3] = \frac{\text{Liquid concentration } [\mu g/mL] * \text{Volume of acetonitrile } [mL]}{\text{Sampling volume } [m^3]}$$
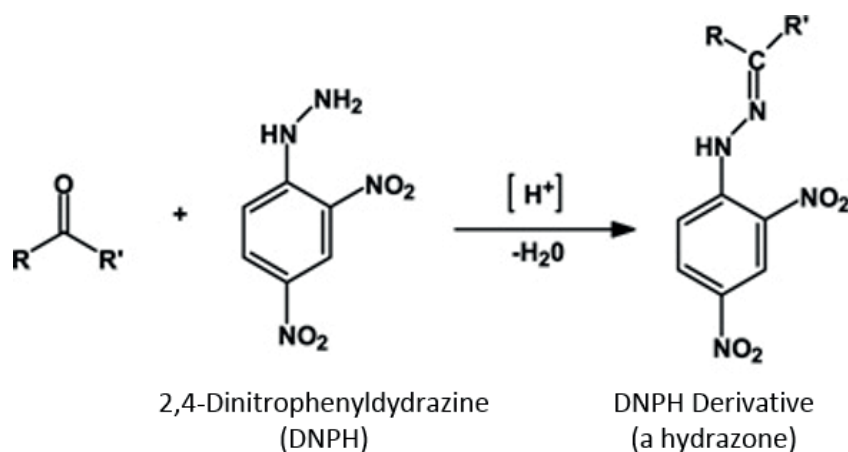
Equation 1

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Figure 4: Reaction process of aldehyde or ketone with 2,4-dinitrophenylhydrazine (DNPH).

Table 3: LODs and LOQs of aldehydes/ketones with the micro-scale chamber-DNPH-HPLC-DAD method (3 L sampling volume, 2 mL ACN).

Aldehyde/Ketone	Liquid concentration		Air concentration	
	LOD [µg/mL] (n=5)	LOQ [µg/mL] (n=5)	LOD [µg/m³] (n=5)	LOQ [µg/m³] (n=5)
Formaldehyde	0.0014	0.0046	0.95	3.05
Acetaldehyde	0.0081	0.0222	5.37	14.81
Acetone	0.1229	0.3684	81.93	245.62
Acrolein	n.d.	n.d.	n.d.	n.d.

HPLC-MS
HPLC-MS detection in SIM mode was also used at the beginning of the study, but the method was only linear up to concentrations of around 0.06 µg/mL. HPLC-MS was found to be more sensitive than HPLC-DAD, but in reality LODs and LOQs of formaldehyde, acetaldehyde and acetone were dictated by the relative high blank values rather than by the sensitivity of the detector. Consequently, a DAD detector was deemed the more the suitable choice for the DNPH method.

Repeatability & Recovery
For validation of the micro-scale chamber method, a 3 µL sample of water containing 0.6 µg each of formaldehyde and acrolein was spiked into a 1 L DHS Large container and 3 L of air subsequently sampled onto a DNPH tube. This recovery experiment was re-peated 5 times using 5 different containers. Average recovery for formaldehyde was found to be 102 % (RSD 6.0 %) when calculated against an HPLC-DAD calibration curve based on direct injection of liquid standard DNPH derivatives into the HPLC system, and 102 % (RSD 6.1 %) when calculated against a micro-scale cham-ber-DNPH-HPLC-DAD calibration curve based on direct injection of a liquid standard into the micro-scale chamber. The recovery of acrolein was calculated to be only 43 % (RSD 10.7 %) based on a direct injection HPLC-DAD calibration curve, but 82 % (RSD 11.7 %) based on a micro-scale chamber-DNPH-HPLC-DAD calibration curve (Table 4). Sampling of acrolein on DNPH cartridges leads to the formation of more than one derivative, whereas the aldehyde/ketone-DNPH standard mix contains only the main acrolein-DNPH derivative [1]. Therefore an underestimation occurs when using the HPLC-DAD calibration.

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Table 4: Recoveries and RSDs (%, n=5) for formaldehyde and acrolein using the micro-scale chamber-DNPH-HPLC-DAD method (3 L sampling, 2 mL ACN elution).

Compound	Amount spiked (µg)	Recovery [%], (n=5)		RSD [%], (n=5)	
		HPLC-DAD calibration	micro-scale chamber-DNPH-HPLC-DAD calibration	HPLC-DAD calibration	micro-scale chamber-DNPH-HPLC-DAD calibration
Formaldehyde	0.6	102	102	6.0	6.1
Acrolein	0.6	43	82	10.7	11.7

Analysis of wood samples
Three different wooden samples (OSD, MDF and plywood) were chosen as test samples and analyzed using the micro-scale chamber-DNPH-HPLC-DAD method. All the board/plate samples were prepared in 6 cm x 9 cm size with front and back surfaces exposed to the chamber. All edges were covered with aluminum foil and the sample was placed vertically in the DHS Large container (Figure 4).
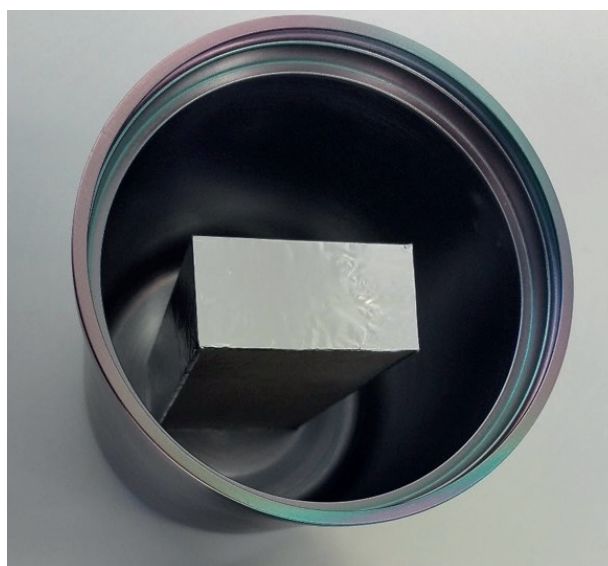
Figure 4: Medium–density fiber board (MDF) prepared in 6 cm x 9 cm size and all edges covered with aluminum foil. The sample was placed vertically in the DHS Large container.

Detected aldehydes/ketones with their calculated air concentrations and surface specific emission rates (SER, in brackets) are listed in Table 5. All samples show emissions of formaldehyde, acetaldehyde and acetone, but no acrolein was found. The OSB sample emitted mainly higher aldehydes, like propionaldehyde, butyraldehyde and (iso)valeraldehyde. The highest formaldehyde and acetone emissions were found for the plywood sample. The average concentration of formaldehyde was 147 µg/m³ with a SER of 41 µg/m²h and an RSD of 8.8 % (n=4). This sample was part of a wooden base plate of a box containing wooden building blocks for children of 1 year and older. Base plates of the same product boxes were tested by the German Stiftung Warentest also showing high amounts of formaldehyde emissions [7]. Although the methods used in these two cases were not the same, the results are comparable.

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Table 5: Air concentrations [µg/m³] and surface specific emission rate (SER [µg/m²h]) obtained from wood plates using micro-scale chamber-DNPH-HPLC-DAD method.

Sample Compound	OSB 12 mm	MDF 12 mm	Plywood* (RSD %, n=4)
Formaldehyde	3 (1)	33 (9)	147 (41, 8.8 %)
Acetaldehyde	24 (7)	26 (7)	13 (4, 16.0 %)
Acetone	< LOQ	< LOQ	312 (87, 8.6 %)
Acrolein	< LDD	< LDD	< LDD
Propionaldehyde	6 (2)	< LDD	< LDD
Butyraldehyde	6 (2)	< LDD	< LDD
(Iso)valeraldehyde	53 (15)	< LDD	< LDD
Hexanal	191 (53)	100 (28)	< LDD

* Plywood is from a base plate of a box containing building blocks for children.

The surface specific emission rate (SER) was calculated using the following equation:

$$SER = c * \frac{N}{L} = c * \frac{N * V}{A}$$

Equation 2

c   analyte concentration in air sample [µg/m³]
N   air exchange rate [3/h]
V   Sampling volume [0.003 m³]
A   Sample surface area [0.0108 m²]
q   Surface specific air flow rate [0.2778 m³/m²h]
L   Loading factor [m²/m3]

Several wooden toys (Figure 5) were also tested using the micro-scale chamber method under the same conditions except that the sizes of the wooden toys varied. The surface area of the samples were measured, surface specific emission rates are shown in Table 6.
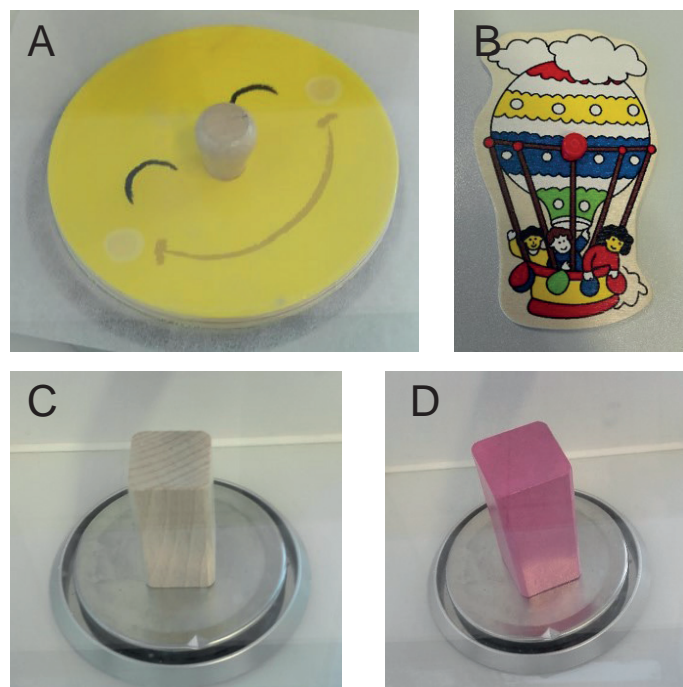

Figure 5: Puzzles (A: "sun" & B: "balloon") and building blocks (C: "nature" & D: "pink").

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Table 6: Air concentrations [µg/m³] and surface specific emission rates (SER [µg/m²h]) obtained from wooden toys using the micro-scale chamber-DNPH-HPLC-DAD method.

Compound	Puzzle "sun" plywood (103 cm²)	Puzzle "balloon" plywood (146 cm²)	Building block „nature" (56 cm²)	Building block "pink" (78 cm²)
Formaldehyde	21 (6)	32 (4)	< LOQ	10 (7)
Acetaldehyde	26 (8)	23 (7)	33 (18)	18 (5)
Acetone	< LOQ	< LOQ	< LOQ	< LOQ
Acrolein	8 (2)	< LOD	< LOD	< LOD
Butyraldehyde	6 (2)	< LOD	< LOD	< LOD
(Iso)valeraldehyde	17 (5)	< LOD	< LOD	< LOD
Hexanal	77(22)	25 (6)	17 (9)	16 (5)

As can be seen in the table, the building block "nature" emitted the lowest amount of formaldehyde (< 3 µg/m³) whereas to the puzzle piece "balloon" made of plywood emitted the highest amount of formaldehyde (32 µg/m³). By contrast, the building block "nature" emitted the highest amount of acetaldehyde (33 µg/m³). Acrolein was detected only in pieces from the puzzle "sun" in a concentration of 8 µg/m³ along with butyraldehyde and (iso)valeraldehyde. The "sun" puzzle piece tested also emitted the highest amount of hexanal (77 µg/m³). The same wooden toys were tested by the German "Stiftung Warentest", which found high levels of formaldehyde emissions in the puzzle piece "balloon", but no formaldehyde in the wood building block "nature" or in the puzzle piece "Sun" [7].

Emission behavior

In order to demonstrate the possibility for automated monitoring of VOC emissions over time from wooden samples, a monitoring program over three days was set up for the MDF sample, alternating between the DNPH-HPLC-DAD and the Tenax-GC-MS methods. Between each DNPH tube sampling (interval at least 5 hours), several Tenax TA samplings were done automatically every hour. As an example, emission profiles for formaldehyde and hexanal obtained using the DNPH-HPLC-DAD method are shown in Figure 5. The emission value of formaldehyde decreases slowly, from 9 µg/m²h after 1 hour under micro-scale chamber sampling conditions (3/h, 23 ± 1 °C, RH = 0 %) to 4 µg/m²h after 72.87 hours under micro-scale chamber sampling conditions (3/h, 23 ± 1 °C, RH = 0 %). Hexanal shows a strong decrease from 28 µg/m²h at 1 hour to 2 µg/m²h at 72.87 hours.
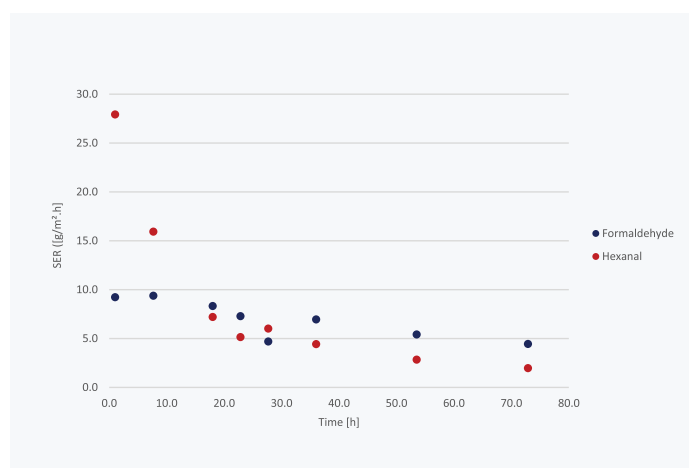
Figure 6: Formaldehyde and hexanal emission profiles of MDF using the DNPH-HPLC-DAD method.

Hexanal emission - DNPH vs. Tenax

The hexanal emission profile obtained with the Tenax-GC-MS method is shown in Figure 7. A pronounced decay was observed, from 104 µg/m²h at 2 hours to 7 µg/m²h at 53.33 hours. The data shows that the DNPH-HPLC method results in a significantly lower hexanal concentration (around one-quarter) compared to the Tenax-GC method. Results are in good accordance with the literature which also reports poor recovery for the analysis of hexanal using DNPH sorbent tubes due to slow derivatization kinetics [8, 9].
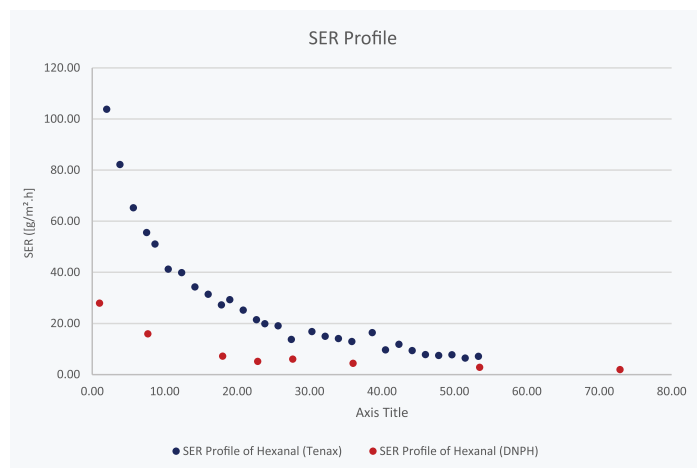
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Figure 7: Hexanal emission profile for MDF using the Tenax-GC-MS method and the DNPH-HPLC method.

TVOCs in toluene equivalents

The total volatile organic compound (TVOC) emission values from n-hexane to n-hexadecane were calculated as toluene equivalents. A decay curve is shown in Figure 8, the SER value of TVOCs decreased from 399 µg/m²h at 2 hour to 209 µg/m²h at 53.33 hours. Using a power function trend line, the TVOC SER values after 3 days (72 hours) and after 28 days (672 hours) were extrapolated and calculated to be 182 µg/m²h and 96 µg/m²h respectively. An estimation of emission values after 3 days or even 28 days with only one or two days of continuous measurements would be a powerful way to study the material emission behavior and therefore shorten the overall test time needed. More tests of different materials would be needed in order to build solid emission model.
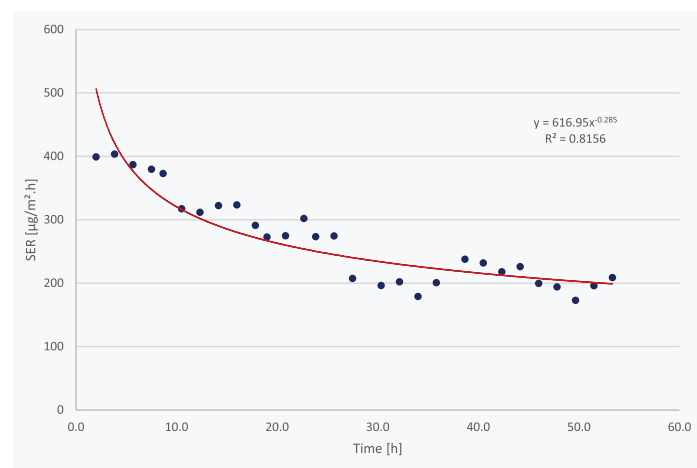

Figure 8: TVOCs emission profile and power trend line for MDF using the Tenax-GC-MS method.

Humidity

It is well known that a higher relative humidity leads to higher formaldehyde emission values [10]. Under low relative humidity, formaldehyde emission values can be underestimated due to a suppressed hydrazine-to-hydrazone conversion [11]. The impact of relative humidity on the emission behavior of formaldehyde in building materials was also studied by Huang et al. [12], the model was built using an airtight chamber according to the c-history method [13] for relative humidity from 20 % to 85 %. Unfortunately, no modeling or relationship was available to estimate the emission value at another relative humidity using the emission value obtained at zero % relative humidity.

Our goal was to quantitatively compare the wood-based products under the same micro-scale chamber conditions and not to achieve the same emission values as those obtained in emission tests in bigger environmental chambers. We have succeeded in developing a time, labor and cost saving screening method for product quality control and development.

Conclusions

This study demonstrates the possibility of combining DNPH derivatization and Tenax TA adsorption, for automated sampling of formaldehyde and other low molecular aldehydes and ketones as well as VOCs in parallel in one micro-scale chamber. VOC analysis via GC-MS was performed automatically after sampling on the same instrument using thermal desorption, while DNPH sorbents were first stored on the instrument and later eluted followed by HPLC-DAD analysis. Automation of the elution step and LC determination is planned.

The micro-scale chamber method using DHS Large containers (1 L) and DNPH sorbent tubes showed high recovery with low RSDs for formaldehyde and acrolein as well as low LODs and LOQs in addition to good linearity. Method applicability was demonstrated by the analysis of several wooden samples.

The DNPH sorbent was found to be suitable for C1-C4 aldehydes, especially for formaldehyde, but not for hexanal and higher aldehydes, a fact that has also been reported by other authors [9]. Acetone showed high and variable background values, probably caused by differing acetone air concentrations in the laboratory. Acrolein forms many derivatives on a DNPH tube, as a consequence calibration needs to be done for the whole sampling process.

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A time and labor saving automated sampling and analysis method was developed that is suitable as screening method for quality control in production processes or for product development. It is also ideal for studying the emission behavior of materials and for building emission models. For example, Xiong et al. have developed a c-history method in a closed chamber to build models in order to calculate the influence of environmental factors (temperature, humidity and air exchange rate) and to compensate for the influence of different chamber types.

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